

(c) recovering said intercalated graphite flake.

22. A method according to claim 21 wherein the graphite flake is contacted with said expansion aid prior to subjecting said graphite flake to electrolytic oxidation.

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23. A method according to claim 21 wherein the graphite flake is contacted with said expansion aid by dissolving said expansion aid in said aqueous intercalant solution prior to subjecting said graphite flake to electrolytic oxidation therein.

24. A method according to claim 21 wherein said expansion aid comprises a carboxylic acid soluble in said aqueous intercalant solution in an amount effective to enhance exfoliation.

25. A method according to claim 24 wherein said carboxylic acid comprises a carboxylic acid selected from the group consisting of lower aliphatic carboxylic acids and dicarboxylic acids and mixtures of these.

26. A method according to claim 25 wherein said acid comprises a carboxylic acid of the formula $H(CH_2)_nCOOH$ wherein n is a number of from 0 to about 5.

27. A method according to claim 21 wherein said intercalant solution contains from about 30 to about 85% water by weight of the solution.

28. A method according to claim 27 wherein said intercalant solution contains from about 50 to about 75% water by weight of the solution.

29. A method according to claim 21 wherein the electrolytic oxidation treatment comprises passing a current between a cathode and the graphite flake as an anode at an anode current density of from about 0.02 to about 0.06 amps per square centimeter.

30. A method according to claim 21, wherein said expansion aid comprises an amount effective to enhance exfoliation of from about 1 to 10% of a carboxylic acid soluble in said aqueous intercalant solution, and said intercalant solution contains from about 30 to about 85% water, all percentages based on the weight of the solution.

31. A method according to claim 21 wherein the electrolytic oxidation treatment comprises passing a current between a cathode and the intercalant wet graphite flake as an anode at a cell voltage of from about 1 to about 6 volts.

32. A method for preparing expandable graphite flake exhibiting one or more improved exfoliation characteristics, which comprises:

(a) contacting graphite flake with an organic expansion aid comprising a carboxylic acid selected from the group consisting of lower aliphatic carboxylic acids and dicarboxylic acids and mixtures of these, said contacting being prior to subjecting said graphite flake to electrolytic treatment;

(b) then, subjecting said graphite flake to an electrolytic oxidation treatment using an aqueous, intercalant solution comprising about 10-75% sulfuric acid to provide intercalated graphite flake, by passing a current through the solution between a cathode and graphite flake wet with the intercalant as an anode at a current density of from about 0.02 to about 0.06 amps per square centimeter and at a cell voltage of from about 1 to about 6 volts; and

(c) recovering said intercalated graphite flake.

33. A method according to claim 32, wherein said expansion aid comprises a carboxylic acid soluble in said aqueous intercalant solution and is employed in an amount of from about 1 to about 10%, both percentages based in the weight of the intercalant solution.

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TECHNICAL FIELD

34. An intercalated graphite flake,
which is contacted with an organic expansion aid, and
which is subjected to electrolytical oxidization with an aqueous
intercalant solution that comprises 10-75% sulfuric acid.

35. The intercalated graphite flake of claim 34, wherein
said expansion aid comprises a carboxylic acid soluble in said
aqueous intercalant solution in an amount effective to enhance
exfoliation.

36. The intercalated graphite flake of claim 34, wherein
said expansion aid comprises an amount effective to enhance
exfoliation of from about 1 to 10% of a carboxylic acid soluble in said
aqueous intercalant solution, and said intercalant solution contains from
about 30 to about 85% water, all percentages based on the weight of the
solution.

37. The intercalated graphite flake of claim 34, wherein
said flake is contacted with an organic expansion aid comprising a
carboxylic acid selected from the group consisting of lower aliphatic
carboxylic acids and dicarboxylic acids and mixtures of these, said

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contacting being prior to subjecting said graphite flake to electrolytic treatment; and then

said flake is subjected to electrolytic oxidation with an aqueous, intercalant solution comprising about 10-75% sulfuric acid by passing a current through the solution between a cathode and graphite flake wet with the intercalant as an anode at a current density of from about 0.02 to about 0.06 amps per square centimeter and at a cell voltage of from about 1 to about 6 volts.

38. The intercalated graphite flake of claim 34, wherein

said flake is subjected to electrolytic oxidation by passing a current through the solution between a cathode and said graphite flake as an anode at a current density of from about 0.02 to about 0.06 amps per square centimeter and at a cell voltage of from about 1 to about 6 volts with said intercalant solution and an organic expansion aid in an amount of from about 1 to 10%.

39. The intercalated graphite flake of claim 34, wherein

said graphite flake is contacted with an organic expansion aid with an intumescent temperature of below about 200°C.

40. The intercalated graphite flake of claim 34, wherein

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